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Interpenetrating Polymer Network (IPN) Adhesives for Electron Beam Cure

by James M. Sands, Steven H. McKnight,
and Bruce K. Fink

ARL-TR-2321 September 2000

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Abstract

Electron beam (e-beam)-processed polymer adhesives have historically performed poorly compared to traditional adhesive technologies due to a lack of toughness engineered into these new types of adhesive materials. Consequently, sequential- and simultaneous-interpenetrating polymer networks (seq-IPN and SIN) were developed and characterized. Seq-IPN adhesive pastes demonstrated exceptional lap-shear strengths (approaching 41 MPa) with glass transition temperatures (T_g) of 100–120° C. The sequential polymerization proceeds by first thermally curing the adhesive to the green-strength. The cure kinetics for the C-staged seq-IPNs have been investigated as a function of temperature and cure acceleration. SIN materials are being investigated to develop durable e-beam-curable film adhesives. The primary advantage of SIN film adhesives, compared to cationic e-beam systems, is insensitivity to surface contaminants (e.g., amine and water), which are known to hinder cure in cationic e-beam polymerization. Preliminary results have shown that the SIN e-beam adhesives have excellent properties. Lap-shear strengths exceeding 27.5 MPa with adhesive T_g approaching 150° C have been demonstrated on primed aluminum substrates. Structural, kinetic, and adhesive performance data for both seq-IPN and SIN structural adhesives cured by e-beam irradiation are presented.

Acknowledgments

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Table of Contents

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List of Figures

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List of Tables

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1. Introduction

Electron beam (e-beam) curing of composite materials is a process technology that will expand performance potential and manufacturing capability for thermoset polymer composites [1] and adhesive bonding. However, the current state-of-the-art (SOA) materials for e-beam processing do not achieve certain resin-dominated properties when compared to autoclave-cured thermosets [2]. A new group of adhesive materials that obtain excellent mechanical performance upon e-beam cure has been developed. These e-beam-cured resins are designed as interpenetrating polymer networks (IPN) [3]. IPNs are polymers that are formed from the independent polymerization of two or more distinct networks, which results in unique molecular and physical microstructures [4]. The final properties of an IPN are very dependent on the composition of the two networks, as well as the polymerization mechanisms and kinetics of each network. Two routes to produce radiation-curable IPN materials are presented in this work. The sequentially cured interpenetrating networks (seq-IPN) presented here are first thermally processed to polymerize one of the networks and form a template matrix for the subsequent e-beam polymerization of the second network. The monomers for the e-beam cure are unaffected by thermal curing and result in a monomer swollen template. Simultaneous interpenetrating networks (SIN) are also studied in this work. The SINs are cured simultaneously during e-beam dose and do not undergo a thermal-staging operations. The monomers for the two networks react independently through noncompeting mechanisms, and the kinetics of each reaction determine the ultimate structure and properties of the final IPN adhesive.

Cationic homopolymerized epoxy-thermosets have been studied as potential e-beam-curable adhesives and composite matrix materials [5]. These systems are brittle [3] and have not produced adhesives that can approach the properties of thermally cured epoxy films or pastes. Various approaches have been evaluated for toughening the cationic systems with moderate success. Yet, adhesive properties are lacking. One issue that has not been thoroughly addressed is the toughening mechanisms for these systems. Traditional epoxy systems can be toughened by inclusion of secondary phase materials (rubbers, thermoplastics). These approaches are

limited in cationic systems due to the different cure mechanisms that limit phase separation and the inability to use nitrogen-containing modifiers. For example, the traditional use of carboxy-terminated-butadiene-nitrile (CTBN) rubbers in toughened epoxy formulations is not possible in cationic systems since the radical cation formed during e-beam processing is poisoned by the presence of lone-pair electrons [6].

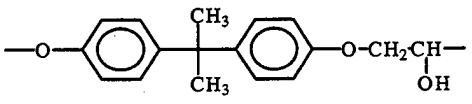
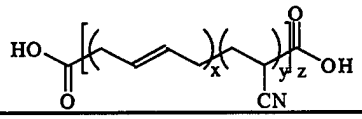
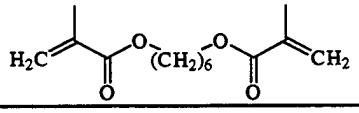
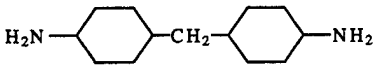
An alternative method of introducing toughness into the e-beam resins includes creating an IPN [7]. Toughness can be improved by appropriate selection of network compositions and polymerization conditions. E-beam-curable seq-IPN structures have been evaluated by Goodman [7]. These systems are comprised of a thermally curable system (epoxy, urethane, etc.) that is polymerized in the presence of free-radical monomers/oligomers. After thermally staging the system, the free-radical species are cross-linked using e-beam irradiation. The epoxy-methacrylate seq-IPN materials have resulted in improved resin properties, including toughness; however, they still do meet adhesive performance requirements.

In this work, these seq-IPNs are modified using CTBN and other elastomer modifiers, as well as chain extenders, to increase the toughness of the materials, with the goal of obtaining useful adhesives for assembly and repair applications. Specifically, the synthesis and properties of elastomer-modified seq-IPN networks are described for simple epoxy-amine (EA) and free-radical methacrylate (FRM) chemistries are described. The development of network structure during epoxy conversion is followed by Fourier-transform infrared spectroscopy (FTIR) for cure temperatures ranging from room temperature to 80° C. The structure of the seq-IPN and the elastomer-modified seq-IPN are examined using atomic force microscopy (AFM) and scanning electron microscopy (SEM). Toughened IPN materials are then formulated as two-part paste adhesives, and the performance of these adhesives is quantified for a number of adherends. Additionally, preliminary data on the performance of simultaneous IPN film adhesives cured by e-beam irradiation is reported.

2. Experimental

2.1 Synthesis of Seq-IPN and Toughened Seq-IPN Materials. The materials selected for the IPNs are DGEBA epoxides and derivatives. The general chemical make-up of the IPNs is shown in Table 1. The seq-IPNs are cured in two stages—a thermal or room temperature cure to C-stage and an e-beam cure—while the SINs are cured by e-beam dose alone. In seq-IPNs, the epoxides are thermally cured with a diamine-curing agent (PACM) to form a template network of EA. The e-beam-cured network is composed of DGEBA-type and hydrocarbon dimethacrylates. E-beam irradiation induces a free-radical generation in the methacrylates, which propagate in the usual radical manner producing the second network (FRM). The cure pathways for these networks are unique and result in the formation of homogeneous EA and FRM networks. The typical reaction scheme for formation of EA by thermal cure and FRM by free radical on e-beam dose to produce seq-IPNs is sketched in Figure 1.

Table 1. Monomer Structures and Chemical Backbone of IPN Formulations

EA Network	Backbone / Structure	FRM Network
DGEBA		M-DGEBA B-DGEBA
ETBN		—
—		HDDMA
1°-DIAMINE		—

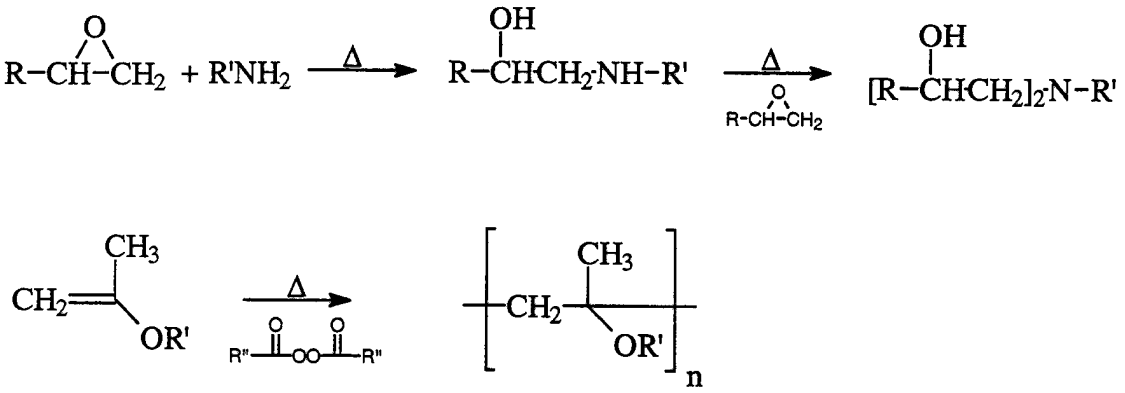


Figure 1. Generic Product Formation in IPN Chemistry.

The SIN materials are composed of roughly equivalent monomers, with the exception of the CTBN. The catalyst for e-beam-induced epoxide curing is the diaryliodonium hexaflouroantimonate (DPI-1) monomer.

A bifunctional coupling additive was synthesized in house to promote bonding of the two independent networks of both types of IPN. The coupling monomer (B-DGEBA) was synthesized by partial methacrylation of diepoxide using methacrylic acid and a catalyst [8]. The degree of coupling between homopolymer networks in the IPNs can be controlled by varying the concentration of the bifunctional monomer in the blend.

Turbidity measurements were used to construct a phase diagram for the elastomer-modified seq-IPN materials. For maximum toughness and minimum impact on modulus and T_g of the network, two-phase materials are desired [9]. Figure 2 shows the epoxy, methacrylate, and elastomer ternary-phase diagram and indicates a window in which phase separation occurs. Adhesive formulations were derived from those compositions that provided phase separation of the rubber domains. The phase separation of the elastomer is further discussed in a later section.

2.2 Kinetics of Cure for Seq-IPNs. The cure kinetics for a down-selected seq-IPN system and a model system were characterized using FTIR spectroscopy for real-time cure analysis

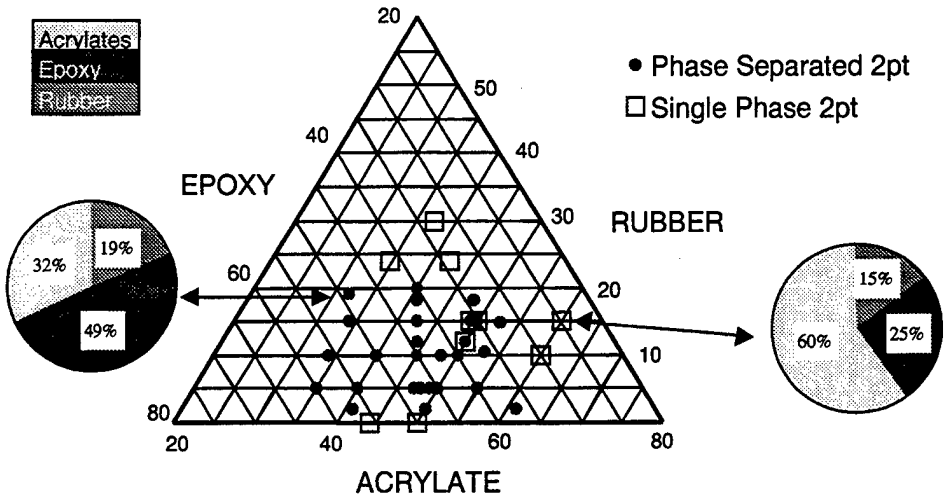


Figure 2. Phase Diagram for IPN Blends Cured to C-Stage at 50° C for 24 hr.

during the C-stage development. Mixed-monomer IPNs can produce complex chemistries on curing. Thus, the cure mechanisms for the seq-IPN adhesive were evaluated using FTIR to track cross reactions as a function of the cure temperature. The resulting conversion vs. time profiles for ADEP01 at various cure-temperatures for the C-stage resin is shown in Figure 3.

In all cases, the conversion to C-stage of the epoxide monomers in the IPN blend is complete for all cure temperatures above the vitrification point (T_v = 36° C) of the formed C-stage. The higher the temperature above the C-stage vitrification, the faster the cure rates. However, high-temperature cure can also result in increased occurrence of amine-methacrylate side reactions, which can diminish the network performance (thermally and mechanically). In our experiments, side reactions were not observed for cure tempeatures below 100° C. Consequently, the reaction scheme for seq-IPN formation is simple enough to facilitate the use of these materials for composite repair and field tests.

Additionally, the IPN can be processed rapidly and at low temperatures by the addition of a catalyst. Figure 4 shows the impact of catalyst on the cure time for ADEP01. A few parts per hundred of catalyst can increase adhesive cure at 50° C to rates equivalent to 80° C cure

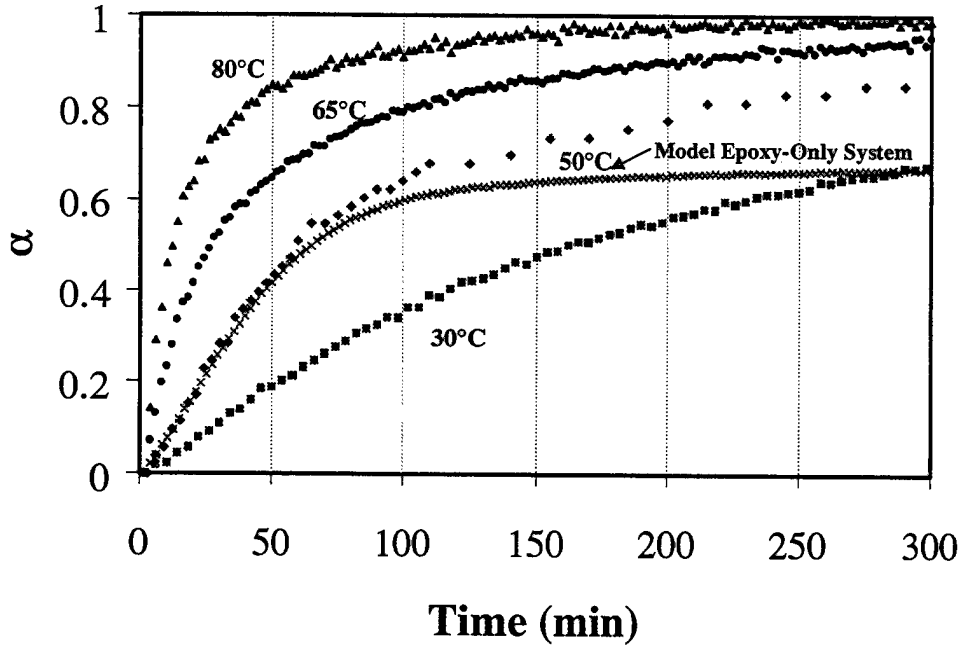


Figure 3. Reaction Progression as a Function of Cure Temperature for C-Stage Formation in IPN Monitored by FTIR.

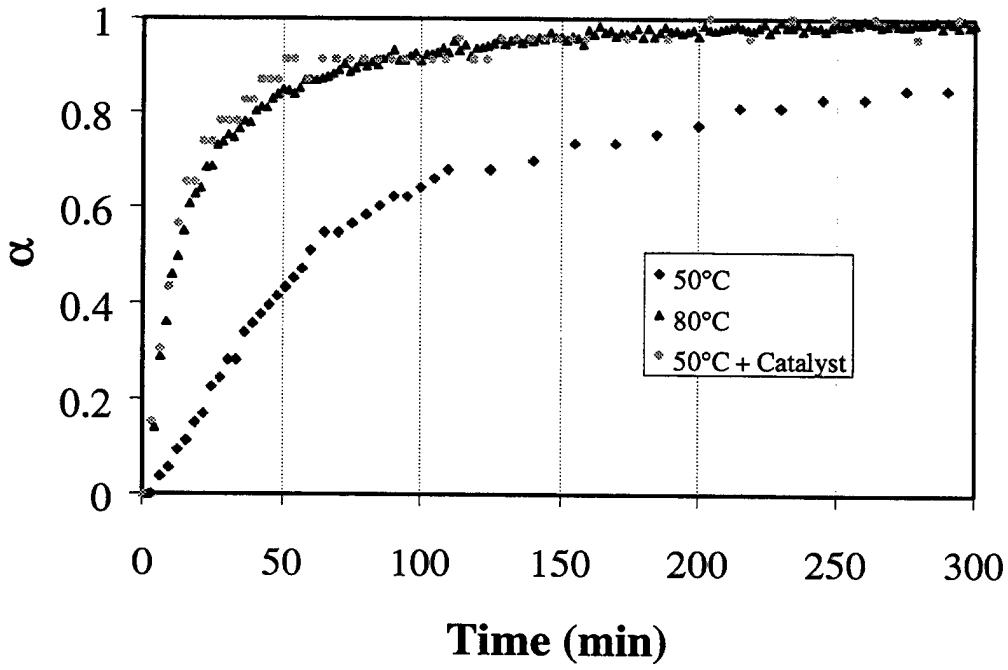


Figure 4. Cure Profile Comparison for IPN With Catalyst to 50° C and 80° C Uncatalyzed Reaction.

reactions. The most significant limitation to catalyzed cure is the onset of the vitrification. Thus, accelerated cure was not evaluated at room temperature for this material.

Due to the rapid cure rate of materials under e-beam processing, cure progression of the materials during e-beam processing is not monitored. Therefore, only final conversions for e-beam materials are presented. The FRM network is unaffected by thermal curing but reacts completely on e-beam processing. The extent of conversion of FRM during e-beam processing is 95% based on calibrated FTIR analysis.

2.3 Structure of Seq-IPN and Toughened Seq-IPN Materials. IPNs are frequently observed to phase- or microphase-separate during polymerization, which greatly influences material properties. The order of the reactions, their rate, and the miscibility of the two networks with one another lead to a number of interesting multiphase morphologies and properties. Upon complete reaction of the EA system, the methacrylates are polymerized using e-beam irradiation.

The materials that were investigated exhibited excellent toughness without substantial modification using rubbers or CTBNs. The toughness of the epoxy-methacrylate IPN was nearly double the values exhibited for each network by itself [10]. The increase of toughness can be attributed to the microphase morphology that was characterized using AFM. Figure 5 shows an AFM image of the IPN microstructure. The small domain size permits significant interactions between the two network structures and results in increased toughness with maintained thermomechanical properties. The toughness of these IPNs can be further increased by the incorporation of the CTBN elastomers. Improved toughness of the virgin resins by 100% was accomplished by the addition of CTBN toughening additives to the baseline IPN material. The phase-separated morphology that accompanies this strategy is shown in the AFM in Figure 6 and the SEM in Figure 7. The discontinuous particles are a CTBN-rich phase that has precipitated during the thermal cure process. No such phase separation is observed for compositions that reside outside the phase envelope shown in Figure 2. A complementary SEM micrograph of a toughened IPN fracture surface is displayed in Figure 7. This picture reveals cavitation around each of the particles, which is consistent with a proposed toughening mechanism for elastomer-modified thermosets [11].

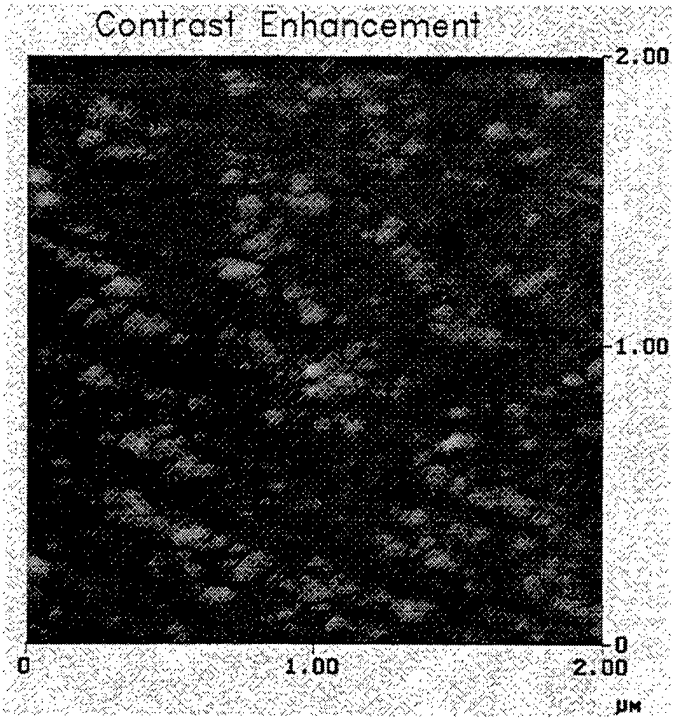


Figure 5. Microphase Structure in Unmodified Epoxy-Methacrylate IPN Materials as Revealed by AFM.

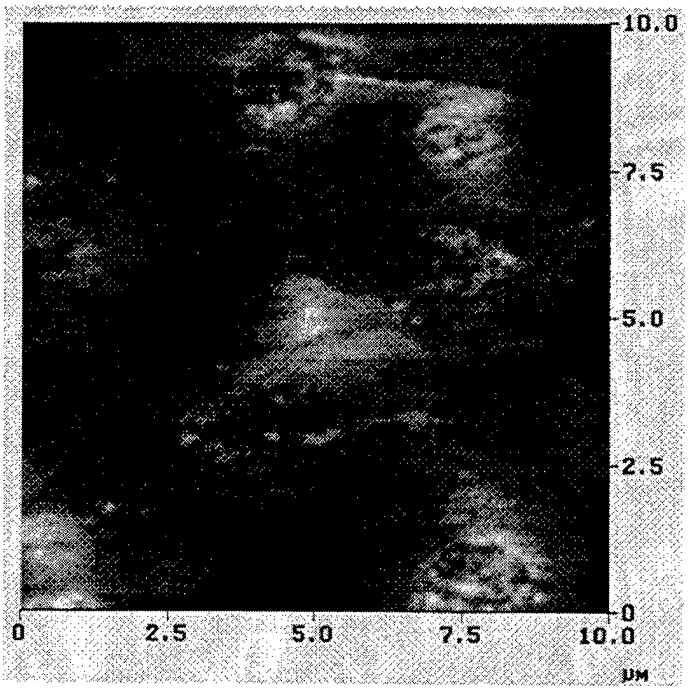


Figure 6. AFM Image Showing the Two-Phase Microstructure in CTBN-Modified Epoxy-Methacrylate IPNs.

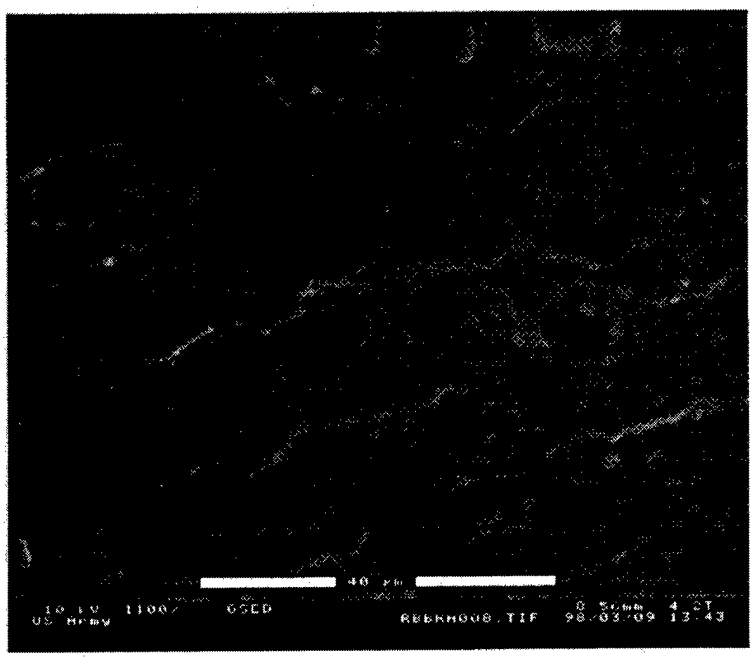


Figure 7. SEM Image That Shows the Two-Phase Microstructure in CTBN-Modified IPNs.

2.4 Mechanical Performance of IPN-Based E-Beam Adhesives. The mechanical performance of the IPNs, formulated as an adhesive, was determined in initial testing by adhesive lap shear strength measurements (ASTM D1002) [12]. The results from early testing of the adhesives in this report are shown in Table 2.

The LSS results are reported for room-temperature tests on all the materials and from elevated temperature analysis for a toughened adhesive paste. The effect of toughening the IPN materials using typical epoxy-network toughness schemes is evident by comparison of LSS performance between ADEP00, the untoughened IPN, and ADEP01. The insertion of rubber particles into the matrix by process-induced phase separation of an epoxy-terminated rubber (ETBN) results in a two-fold increase in LSS, while thermal performance is equivalent to the nonmodified system. One can attribute the improved performance to the invocation of alternate energy absorbing modes introduced into the matrix, including shear banding and rubber cavitation [13]. These modes are evident by observing the fracture surface of the adhesive by SEM shown in Figure 7. The results in Table 2 also demonstrate that the proper design of

Table 2. LSS and T_g Comparisons for Seq-IPN and Commercial Adhesives

Substrate and Surface Preparation	Test Temp (°C)	ADEP00 Avg. ± std (MPa)	ADEP01 Avg. ± std (MPa)	JCAT1 Avg. ± std (MPa)	FM73 Avg. ± std (MPa)	EA9394 Avg. ± std (MPa)
Al 7075/P2	25	20.6 ± 4	36.5 ± 4	NT	39.9 ± 1	19.3 ± 2
Al 7075/P2 (green)	25	8.2 ± 2	13.1 ± 2	NA	NA	NA
Al 2024/Br127	25	NT	32.8 ± 4	28.2 ± 2	43.9 ± 1	28.9
Al 2024/Br127	104	NT	28.9 ± 1	NT	28.5 ± 1	17.9
3501-6	25	NT	22.7 ± 4	NT	NT	26.0 ± 2
977-3	25	NT	33.4 ± 5	NT	38.1 ± 2	NT
T_g(°C) Dry	—	123	121	150	95	65
Form	—	Paste	Paste	Unsupported Film	Film	Paste

IPN adhesive materials for e-beam cure can result in high-performance adhesive properties. Although this study targets moderate thermal performances (T_g > 110° C) pastes, the LSS of the ADEP01 is comparable to commercially available film adhesives such as Cytec's FM73 on both composite and aluminum adherends. Based upon the performance of this model system, the ADEP01 paste chemistry was selected for additional testing and adhesive formulation modification by Scientific Research Laboratories (SRL) to explore the impact of filler additives on the adhesive performance. SRL reported the results of these filler studies [14].

The SIN adhesives have just entered development. Initial formulation of SINs resulted in the JCAT1 adhesive for film applications. Formulation of JCAT1 includes the DPI-1 cationic initiator. Consequently, the JCAT1 series is subject to typical poisonings associated with cationic-cured materials. However, the JCAT1 bonding performance on BR127 primer, a common amine-based primer, was evaluated and achieved surprising results. The final SIN that was produced possessed a relatively high T_g material, with respectable room-temperature LSS performance. These results are also presented in Table 2.

Although SIN development is underway, it appears that SIN adhesives can dramatically improve the stability of the film adhesives and improve the resistance to catalyst poisoning associated with cationic systems presented previously.

3. Conclusions

The development of e-beam processible adhesives demands a novel approach to designing adhesive materials, including the development of new materials technologies for advanced applications. By pursuing e-beam materials developments for ground vehicle applications, the strict performance criteria associated with aerospace applications is avoided. The Army has demonstrated that e-beam processible adhesives can be developed, which provide excellent LSS performance. These high-performance materials, which will adequately meet many military requirements for composite and adhesive specifications, provide a positive framework from which out-of-autoclave processing technology can proceed.

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Electron beam (e-beam)-processed polymer adhesives have historically performed poorly compared to traditional adhesive technologies due to a lack of toughness engineered into these new types of adhesive materials. Consequently, sequential- and simultaneous-interpenetrating polymer networks (seq-IPN and SIN) were developed and characterized. Seq-IPN adhesive pastes demonstrated exceptional lap-shear strengths (approaching 41 MPa) with glass transition temperatures (T_g) of 100–120° C. The sequential polymerization proceeds by first thermally curing the adhesive to the green-strength. The cure kinetics for the C-staged seq-IPNs have been investigated as a function of temperature and cure acceleration. SIN materials are being investigated to develop durable e-beam-curable film adhesives. The primary advantage of SIN film adhesives, compared to cationic e-beam systems, is insensitivity to surface contaminants (e.g., amine and water), which are known to hinder cure in cationic e-beam polymerization. Preliminary results have shown that the SIN e-beam adhesives have excellent properties. Lap-shear strengths exceeding 27.5 MPa with adhesive T_g approaching 150° C have been demonstrated on primed aluminum substrates. Structural, kinetic, and adhesive performance data for both seq-IPN and SIN structural adhesives cured by e-beam irradiation are presented.

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